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14. ABSTRACT
Earlier project activities demonstrated that half-face respirators integrated with fibrillar polymer structures provide much higher overall fit factors (FFoverall) against challenging facial skin conditions (including unshaved, wet and wet-unshaved) when compared with a conventional (non-modified) half-mask elastomeric respirator. The previous report covered the initiation of comprehensive evaluation of polyurethane microfibrillar arrays integrated with half-face respirator mask using the NIOSH 25-subject bivariate panel; this effort was continued into this reporting

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BIO-INSPIRED DRY ADHESIVES

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The immediate plans in project emphasize: (i) evaluation of the scalability and commercial merits of mask respirators with bio-inspired adhesive integrated into their peripheral seals; and (ii) assessment of the competitive position of the new bio-inspired adhesives in broader fields of application.

BIO-INSPIRED DRY ADHESIVES

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Earlier project activities demonstrated that half-face respirators integrated with fibrillar polymer structures provide much higher overall fit factors ($FF_{overall}$) against challenging facial skin conditions (including unshaved, wet and wet-unshaved) when compared with a conventional (non-modified) half-mask elastomeric respirator. The previous report covered the initiation of comprehensive evaluation of polyurethane microfibrillar arrays integrated with half-face respirator mask using the NIOSH 25-subject bivariate panel; this effort was continued into this reporting period. So far, a full panel of 25 subjects have been medically cleared and tested with modified half-face respirator masks. This report covers fit testing of the 25-subjects, and presents detailed test data and their analysis.

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PROGRESS VERSUS TIME SCHEDULE

The progress towards accomplishing the project tasks is presented below in the context of the overall time schedule of the project.

MILESTONES	*MONTHS*							
	1-6	7-12	13-18	19-24	25-27	28-30	31-33	34-36
1. Develop securing and sealing requirements	c							
2. Refine the theoretical models of bio-inspired adhesives		c						
3. Identify theoretically viable ranges of design parameters			c					
4. Refine the synthetic skin			c					
5. Refine the processing conditions of fibrillar arrays			c					
6. Optimization of the bio-inspired adhesive design						c		
7. Assess the adhesion qualities of adhesives against rough skin						c		
8. Assess the performance characteristics of adhesives				i	i	i	x	
9. Develop fabrication techniques for full-facepiece respirator				i	i	i	x	
10. Evaluate and improve respirators through tests on human subjects					i	i	x	
11. Assess performance advantages of new adhesives on respirators								x
12. Evaluate the scalability and commercial merits								x

i = in progress c = completed x = planned completion

Introduction

Bio-inspired adhesives provide strong adhesion by utilizing micro- and nano-structures to overcome surface roughness and establish thorough contact with the substrate. Bio-inspired adhesivers, however, still have leakage problems. Biological adhesion mechanisms have evolved in applications where sealing is not a concern; biomimetic adhesives are not, therefore, inherently leak-resistant. Permeation can occur through the generally networked pore system occurring within biological adhesives (Figure 1a), compromising their sealing qualities. This project has so far developed a unique design of bio-inspired adhesives to enhance the sealing qualities of polymeric fibrillar structures (bio-inspired adhesives) through development of micro-patterned structure (e.g., by incorporating continuous micro-ribbons around fibrillar regions, as shown in Figure 1b. Previous work demonstrated half-face respirators integrated with these fibrillar polymer structures provided much higher overall fit factors ($FF_{overall}$) against challenging facial skin conditions, including unshaved, wet and wet-unshaved faces, when compared with a conventional (non-modified) half-mask elastomeric respirator. This report covers a comprehensive evaluation of half-face respirator masks modified with bio-inspired adhesive against 25 subjects of the NIOSH bivariate panel, with due consideration given to their facial dimensions (Figure 2). Each subject was tested while wearing the prototype half-face respirator mask modified with bio-inspired adhesvie as well as an identical conventional half-face respirator mask (with no peripheral modification) used as control.

In this phase of the project, a total of 120 subjects were identified for initial screening. The selected ones have been subjected to measurement of facial dimensions and the medical clearance process. Those who met the requirements of the investigation were fit-tested using a TSI Portacount. The final tested cohort included 17 male and 8 female subjects; among them, 13 were Caucasians, 8 Asians, and 4 African Americans/African origin. All subjects tested in this phase were well-shaved, and had dry face surfaces.

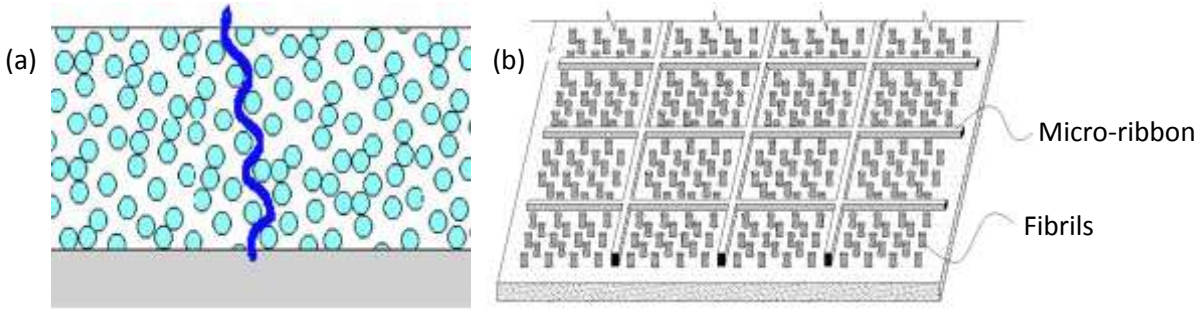


Figure 1. Permeation Through the Networked Pore System within Fibrillar Bio-inspired Adhesive Structures (a); and Control of Permeation by Introducing Micro-Ribbons Separating Fibrillar Regions.

Experimental Program

Fabrication of Microfibrillar Adhesives, and Assembly into Masks

The required quantities of bio-inspired adhesive strips based on ST-3040 polyurethane (PU), with 2 cm width and 10 cm length, were produced at a controlled thickness using a silicon template fabricated via photolithography, as shown in Figure 3. The micro-patterned structure had one continuous micro-ribbon and 60 rows of microfibrils. No further treatments were performed on these micro-patterned structures. The strips were manually assembled onto half-face respirator masks of various sizes (S, M, and L) (Figure 4). The respirator masks used here were 3M 6200-07025; these half-face, reusable respirators are recognized for offering reliable and convenient respiratory protection; per NIOSH approvals, they can provide protection, in different circumstances, against particulates and a wide variety of gases and vapors. In order to assemble the fibrillar polymer adhesive onto the peripheral rubbery material, a silicone adhesive transfer tape was attached on the rubbery material; the dry adhesive strips with same thickness were then assembled onto the mask. The small gaps between adhesive strips were sealed using an acrylic PSA. Figure 4 (right) shows the half-face respirator integrated with bio-inspired adhesive strips.

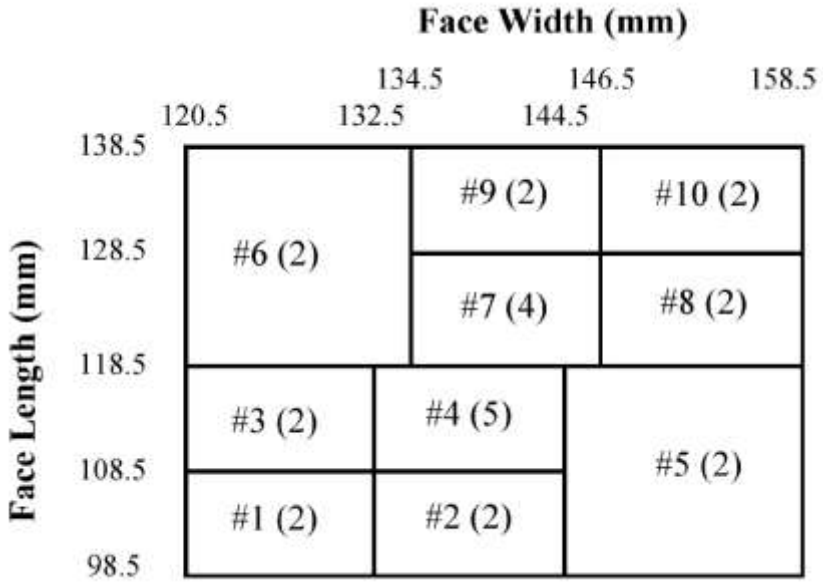
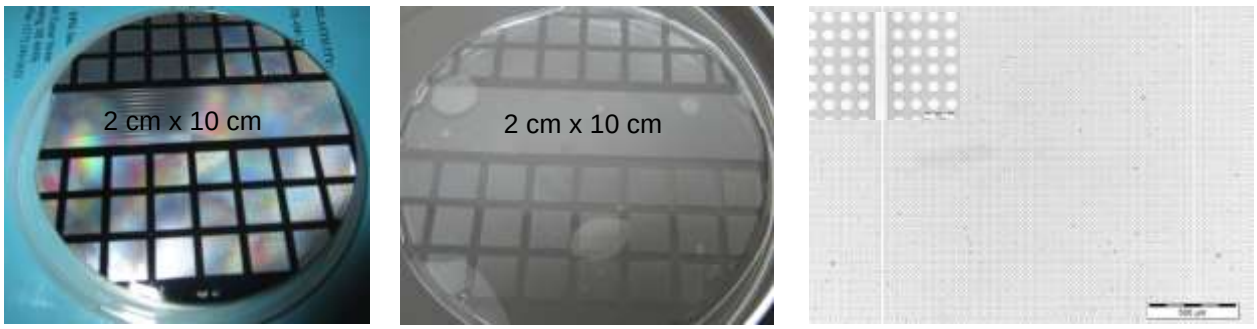


Figure 2. The NIOSH 25-Subject Bivariate Panel.



(a) (b) (c)

Figure 3. Fabrication of Bio-Inspired Adhesives for Integration with Half-Face Respirator Masks: (a) Silicon Template; (b) PU Prototype; and (c) Optic Microscope Image of the Prototype.

Fit Testing of Respirator Masks

A standard fit test program was conducted using a TSI Portacount. The test respirator was donned on each selected human subject who performed a series of exercises following the standard OSHA fit testing protocol, including normal breathing, deep breathing, turning head side-to-side and up-and-down, etc. (total of seven exercises). A particle concentration of NaCl challenge aerosol was measured outside (C_{out}) and inside (C_{in}) of the tested respirator, and a fit factor, FF, was determined as a ratio of above concentrations: C_{out}/ C_{in} (inverse of penetration, P). The FF-value was determined for each exercise. The overall fit factor was determined using the following equation:

$$FF_{overall} = \frac{1}{\sum_{i=1}^{7} \frac{1}{FF_i}},$$

where, FF_i is an exercise-specific fit factor. Each manufactured respirator prototype was tested once. Following such a test, a non-modified respirator (3M 6200-07025 half-mask) was tested on each subject for comparison. The geometric mean and geometric standard deviation for three replicated measurements of FF were calculated for the non-modified respirator.

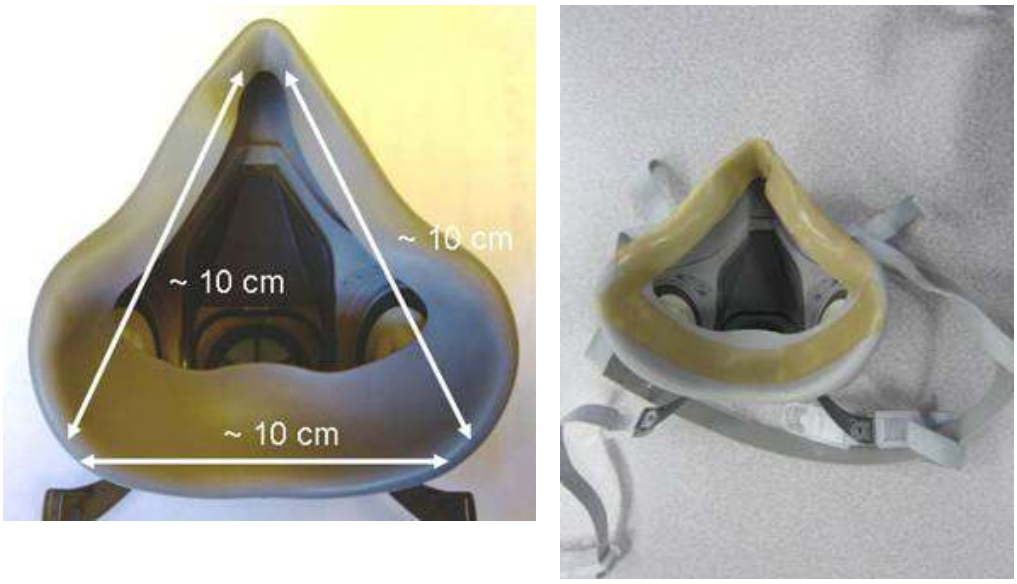


Figure 4. Half-Face Mask Without (left) and With (right) Bio-Inspired Adhesive.

Test Results and Discussion

Results of fit tests on 25 subjects of the NIOSH bivariate panel are summarized in Tables 1 and 2, and Figure 5. The overall fit factors (FFs) determined for PU (ST3040) Half Mask ranged from 159 (Subject #T006) to 57,700 (Subject #T011), with geometric mean (GM) of 7,907, and geometric standard deviation (GSD) of 4.9. All overall FF values for ST3040 were above 100 (a notable threshold). Only 20% of subjects exhibited $FF_{overall}$ <3,000; FF = 3,000 translates into a 99.97% protection. Given that the tested elastomeric respirator is equipped with P100 filters having 99.97% collection efficiency, an FF-value in excess of 3,000 suggests no or negligibly small face-seal leakage. For a vast majority of tested subjects, the PU Half Mask prototype offered an exceptionally good protection with $FF_{overall}$ >10,000. Two subjects (#T06 and #T09) exhibited $FF_{overall}$ values between 100 and 1000, which were about two orders of magnitude below those of the rest of panel.

The non-modified (control) respirator showed overall FFs between 37 and 92,800, featuring GM = 4,779 and GSD = 9.1. The range and variability of values obtained with non-modified respirator were considerably greater than those determined for ST3040. When wearing the conventional Half Mask, one subject had $FF_{overall}$ below 100, and six subjects had $FF_{overall}$ between 100 and 1,000. Ten subjects (40%) had $FF_{overall}$ below 3,000, suggesting that face-seal leakage was present in twice greater number of subjects wearing the conventional Half Mask as compared to those tested with the ST3040 prototype.

Although the ST3040 Half Mask was found advantageous over the control respirator, as substantiated above, the paired t-test fell short of proving that the difference was

statistically significant ($p>0.05$). We attributed this finding to very high variability of data, especially for the control data set.

In addition to the overall fit factor, seven exercise-specific fit factors were determined for each subject in each experiment (see Tables 1 and 2). This data and the similar set obtained with the control elastomeric respirator are being analyzed. Additionally, since both the exercise-specific and overall fit factors obtained with the tested respirators may be affected by facial dimensions, various data analyses are being performed to explore if a meaningful relationship can be established. The preliminary review suggests that performance of ST3040 respirator is more consistent (in terms of between-exercise variability as well as between-subject variability) than that of the conventional respirator.

In many tests conducted with ST3040, only few particles were detected inside the respirator during certain fit testing exercises. While a low particle reading inside the respirator translates into extremely high FFs (a positive finding), we acknowledge that "shortage" of the counted particles also represented a notable experimental challenge, because high data variability becomes unavoidable for respirators of such high protection efficiency.

CONCLUSIONS AND IMMEDIATE PLANS

In this reporting period, the NIOSH 25-subject bivariate panel has been medically cleared and tested with bio-inspired adhesive-modified half-face respirator. The overall fit factors (FFs) determined for masks modified with bio-inspired adhesives ranged from 159 to 57,700, with a geometric mean (GM) of 7,907, and geometric standard deviation (GSD) of 4.9. The non-modified (control) respirator provided overall FFs between 37 and 92,800, featuring GM of 4,779 and GSD of 9.1. In addition to the overall fit factor, seven exercise-specific fit factors indicated that the performance of modified respirator mask is more consistent (in terms of between-exercise variability as well as between-subject variability) than that of the conventional respirator.

The immediate plans in project emphasize: (i) evaluation of the scalability and commercial merits of respirator masks with peripheral seals modified using bio-inspired adhesive; and (ii) assessment of the competitive position of the new bio-inspired adhesives in broader fields of application.

Table 1. Exercise-specific and overall fit factors obtained for the ST3040 Half Mask prototype (Technova Corporation) donned on 25 subjects (well shaved, dry skin).

ID	Type	$FF^{[1]}_{normal}$	FF_{deep}	$FF_{side\text{-}side}$	$FF_{up\text{-}down}$	FF_{talk}	FF_{bend}	$FF^{[2]}_{normal}$	$FF_{overall}$
T001	ST3040	339000	43700	999000	536000	4580	66900	999000	**26600**
T002	ST3040	104000	65800	252000	23000	11200	30600	103000	**34300**
T003	ST3040	24800	15600	45000	101000	3510	63800	599000	**15900**
T004	ST3040	27900	39100	26900	3760	3710	11500	193000	**9630**
T005	ST3040	54900	73000	6370	243000	29600	38100	590000	**27400**
T006	ST3040	3780	11300	49	62	143	12400	66600	**159**
T007	ST3040	57600	38100	85500	109000	10600	189000	316000	**41800**
T008	ST3040	48200	237000	181	11100	6420	467000	999000	**1200**
T009	ST3040	220	6920	72	471	2680	1130	9570	**317**
T010	ST3040	36800	28400	22700	90900	7720	76900	255000	**26500**
T011	ST3040	34900	125000	334000	193000	14400	249000	335000	**57700**
T012	ST3040	13800	7590	53300	14300	7670	14600	54100	**13700**
T013	ST3040	16200	27300	52000	30000	7780	84000	450000	**23800**
T014	ST3040	38800	34700	26000	27800	817	27500	86000	**4990**
T015	ST3040	108000	35300	18100	1710	2680	138	186000	**843**
T016	ST3040	4430	12500	23200	21300	2900	68400	110000	**9150**
T017	ST3040	21300	9590	9040	21100	5190	13500	102000	**11900**
T018	ST3040	19800	26100	90800	88700	10000	44300	77300	**28300**
T019	ST3040	35100	61600	131000	285000	22300	488	83800	**3230**
T020	ST3040	16200	39600	51400	51000	4960	45600	144000	**19600**
T021	ST3040	147000	266000	302000	38500	8690	117000	530000	**42300**
T022	ST3040	11400	27700	14900	18600	12600	49300	52800	**19200**
T023	ST3040	9830	5010	5840	5830	1590	6300	25200	**4750**
T024	ST3040	27200	79900	292	822	6150	631	353000	**1080**
T025	ST3040	21500	25100	146000	141000	1510	511	737	**1710**

Table 2. Exercise-specific and overall fit factors obtained for the conventional Half Mask elastomeric respirator (control) donned on 25 subjects (well shaved, dry skin).

ID	Type	FF^{1}_{normal}	FF_{deep}	$FF_{side\text{-}side}$	$FF_{up\text{-}down}$	FF_{talk}	FF_{bend}	FF^{2}_{normal}	$FF_{overall}$
T001	CONTROL	160	137	329	312	151	207	236	**197**
T002	CONTROL	737	1370	712	1730	11700	120	1230	**526**
T003	CONTROL	20100	63800	55800	62600	9940	11800	240000	**24200**
T004	CONTROL	13000	19200	40100	34700	5260	91600	355000	**18100**
T005	CONTROL	28100	46900	9400	47700	20700	14000	215000	**22600**
T006	CONTROL	4480	18900	60	82	84	18800	78600	**170**
T007	CONTROL	34800	19600	62200	90200	5240	91300	147000	**22100**
T008	CONTROL	277000	536000	961000	417000	9780	279000	611000	**60100**
T009	CONTROL	4810	12000	88	1440	689	897	1320	**446**
T010	CONTROL	140000	276000	53	6	2180	50	29600	**37**
T011	CONTROL	86300	227000	263000	345000	20800	64700	947000	**80100**
T012	CONTROL	297000	117000	319000	999000	22300	79100	535000	**92800**
T013	CONTROL	14600	22800	38100	57400	10200	126000	284000	**26300**
T014	CONTROL	112000	85000	92800	111000	2190	195000	365000	**13800**
T015	CONTROL	50500	32500	1220	27200	2500	126000	58400	**5250**
T016	CONTROL	12100	57000	19100	1970	10700	9830	268000	**8140**
T017	CONTROL	7440	15800	11100	33900	965	9250	62000	**4730**
T018	CONTROL	47000	30100	299000	307000	18800	35000	276000	**47700**
T019	CONTROL	17000	104000	54400	197000	17200	445	36700	**2880**
T020	CONTROL	274000	236000	556000	636000	7820	12000	341000	**31000**
T021	CONTROL	55200	142000	92900	156000	6720	364	203000	**2370**
T022	CONTROL	51700	37600	22500	16600	12900	21700	35300	**23100**
T023	CONTROL	7270	12000	2660	30400	714	947	103000	**2260**
T024	CONTROL	914	41000	175	772	6330	61500	10500	**834**
T025	CONTROL	1010	2670	563	30	34	5370	127000	**105**

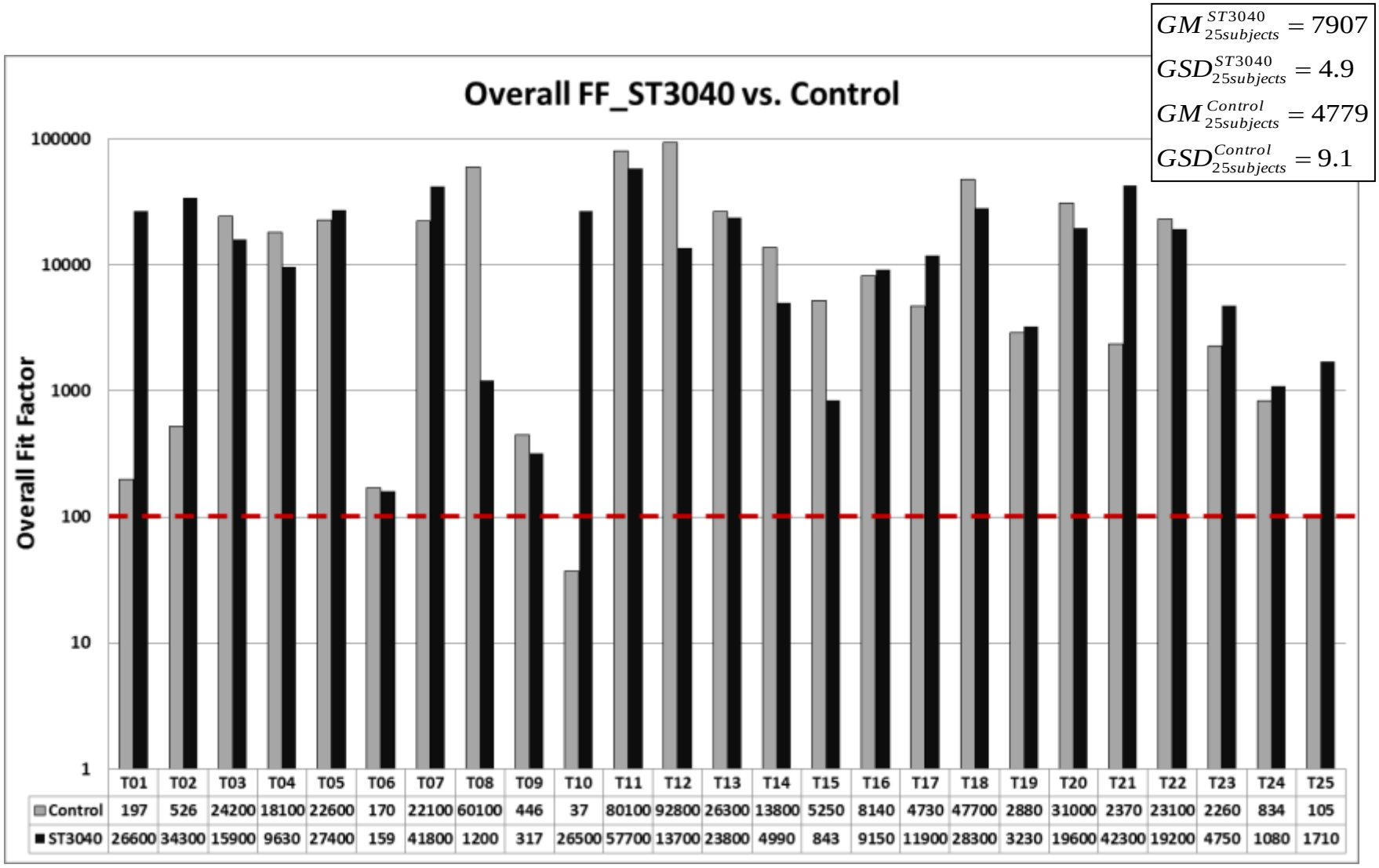


Figure 5. Overall fit factors determined for 25 subjects (well shaved, dry skin) wearing the ST3040 prototype and a conventional Half Mask elastomeric respirator (control).